

Quality Control Checks for BTEX/GRO

Method: 8015D, Volatile nonhalogenated organics by GC/FID

Matrix: Water

Reporting Limit: 20 µg/L

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Initial Calibration (ICAL)	ICAL prior to sample analysis.	$RSD \leq 20\%$; $r^2 \geq 0.990$	Correct problem then repeat ICAL.	6 standards ranging from 20 µg/L to 5000 µg/L
Second source calibration verification (ICV)	Once after each ICAL.	% recovery $\pm 20\%$	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL	Preferably out-of-house, critiqued standard or else a standard from different lot than calibration standards
Continuing Calibration Verification (CCV)	<u>GC/MS</u> : Daily before sample analysis and every 12 hours of analysis time. <u>GC</u> : Every 10 samples and at end of run	% recovery $\pm 20\%$	Restandardize and rerun all samples from last acceptable QC or check sample	Approximate mid-range standard made from working standards stock
Continuing Calibration Blank (CCB)	N/A	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Method Blank (BLK)	Once per run	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Preparation Blank (BLK)	With each preparation batch	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other sample analysis.
Laboratory Control Sample (LCS) Also known as blank spike (BS)	Once per batch or every 20 samples	Spike recovery within statistical limits or $\pm 30\%$	Same as matrix spike	DI water fortified with the same spike as the MS/MSD

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Matrix Spike (MS)	Once per batch or every 20 samples	Spike recovery same as LCS	Check for instrument drift, respoke, and retest	Fortified unknown sample
Matrix Spike Duplicate (MSD)	Every MS	Spike recovery same as MS; RPD \leq 25% between MS and MSD	Same as matrix spike	Duplicate MS
Detection Limit Standard (CRL)	N/A	\pm 50% of expected value	Correct instrument's sensitivity problem or redetermine and raise reporting limits	Low level standard at the RL
Surrogate spike	Every field sample, standard, and QC sample.	Recovery \pm 30%	For QC and field samples, correct problem then reprep and reanalyze all failed samples with failed surrogates, if sufficient sample material is available.	If obvious chromatographic interference with surrogate is present, reanalysis is not necessary.
Standard Reference Material (SRM)	Once per batch (minimum)	Published limits or \pm 30% of true value		May be used in place of an LCS
Internal standards verification (GC/MS)	Every field sample, standard, and QC sample.	N/A	Inspect mass spectrometer and GC for malfunctions.	
Tuning (GC/MS)	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify.	

Sample Preparation Summary

Preparation Method: 5030C

Sample Amount: 5.0 mL

Final Volume: 5.0 mL

Quality Control Checks for TPH/DRO

Method: 8015D, Nonvolatile nonhalogenated organics by GC/FID

Matrix: Water

Reporting Limit: 20 µg/L

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Initial Calibration (ICAL)	ICAL prior to sample analysis.	$RSD \leq 20\%$; $r^2 \geq 0.990$	Correct problem then repeat ICAL.	6 standards ranging from 20 µg/mL to 500 µg/mL
Second source calibration verification (ICV)	Once after each ICAL.	% recovery $\pm 20\%$	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL	Preferably out-of-house, critiqued standard or else a standard from different lot than calibration standards
Continuing Calibration Verification (CCV)	<u>GC/MS</u> : Daily before sample analysis and every 12 hours of analysis time. <u>GC</u> : Every 10 samples and at end of run	% recovery $\pm 20\%$	Restandardize and rerun all samples from last acceptable QC or check sample	Approximate mid-range standard made from working standards stock
Continuing Calibration Blank (CCB)	N/A	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Method Blank (BLK)	Once per run	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Preparation Blank (BLK)	With each preparation batch	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other sample analysis.
Laboratory Control Sample (LCS) Also known as blank spike (BS)	Once per batch or every 20 samples	Spike recovery within statistical limits or $\pm 30\%$	Same as matrix spike	DI water fortified with the same spike as the MS/MSD

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Matrix Spike (MS)	Once per batch or every 20 samples	Spike recovery same as LCS	Check for instrument drift, respoke, and retest	Fortified unknown sample
Matrix Spike Duplicate (MSD)	Every MS	Spike recovery same as MS; RPD \leq 25% between MS and MSD	Same as matrix spike	Duplicate MS
Detection Limit Standard (CRL)	N/A	\pm 50% of expected value	Correct instrument's sensitivity problem or redetermine and raise reporting limits	Low level standard at the RL
Surrogate spike	Every field sample, standard, and QC sample.	Recovery \pm 40%	For QC and field samples, correct problem then reprep and reanalyze all failed samples with failed surrogates, if sufficient sample material is available.	If obvious chromatographic interference with surrogate is present, reanalysis is not necessary.
Standard Reference Material (SRM)	Once per batch (minimum)	Published limits or \pm 30% of true value		May be used in place of an LCS
Internal standards verification (GC/MS)	Every field sample, standard, and QC sample.	N/A	Inspect mass spectrometer and GC for malfunctions.	
Tuning (GC/MS)	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify.	

Sample Preparation Summary

Preparation Method: 3520C
Sample Amount: 1000 mL
Final Volume: 1.0 mL

Quality Control Checks for VOCs

Method: 8260C, Volatile organics by GC/MS with Selective Ion Monitoring (SIM)

Matrix: Water

Reporting Limit: 0.2 µg/L

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Initial Calibration (ICAL)	ICAL prior to sample analysis.	$RSD \leq 20\%$; $r^2 \geq 0.990$	Correct problem then repeat ICAL.	7 standards ranging from 0.2 µg/L to 10 µg/L
Second source calibration verification (ICV)	Once after each ICAL.	% recovery $\pm 20\%$	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL	Preferably out-of-house, critiqued standard or else a standard from different lot than calibration standards
Continuing Calibration Verification (CCV)	<u>GC/MS</u> : Daily before sample analysis and every 12 hours of analysis time. <u>GC</u> : Every 10 samples and at end of run	% recovery $\pm 20\%$	Restandardize and rerun all samples from last acceptable QC or check sample	Approximate mid-range standard made from working standards stock
Continuing Calibration Blank (CCB)	Once to check instrument, then after every CCV	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Method Blank (BLK)	Once per run	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Preparation Blank (BLK)	With each preparation batch	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other sample analysis.
Laboratory Control Sample (LCS) Also known as blank spike (BS)	Once per batch or every 20 samples	Spike recovery within statistical limits from DoD LCS Study	Same as matrix spike	DI water fortified with the same spike as the MS/MSD

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Matrix Spike (MS)	Once per batch or every 20 samples	Spike recovery same as LCS	Check for instrument drift, respire, and retest	Fortified unknown sample
Matrix Spike Duplicate (MSD)	Every MS	Spike recovery same as MS; RPD \leq 30% between MS and MSD	Same as matrix spike	Duplicate MS
Detection Limit Standard (CRL)	Once per run	\pm 50% of expected value	Correct instrument's sensitivity problem or redetermine and raise reporting limits	Low level standard at the RL
Surrogate spike	Every field sample, standard, and QC sample.	Statistical limits from DoD LCS Study	For QC and field samples, correct problem then reprep and reanalyze all failed samples with failed surrogates, if sufficient sample material is available.	If obvious chromatographic interference with surrogate is present, reanalysis is not be necessary.
Standard Reference Material (SRM)	Once per batch (minimum)	N/A		May be used in place of an LCS
Internal standards verification (GC/MS)	Every field sample, standard, and QC sample.	EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions.	
Tuning (GC/MS)	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify.	

Sample Preparation Summary

Preparation Method: 5030C

Sample Amount: 25 mL (large volume)

Final Volume: 25 mL

Quality Control Checks for SVOCs

Method: 8270D, Semivolatile organics by GC/MS

Matrix: Water

Reporting Limit: 0.1 µg/L

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Initial Calibration (ICAL)	ICAL prior to sample analysis.	$RSD \leq 20\%$; $r^2 \geq 0.990$	Correct problem then repeat ICAL.	9 standards ranging from 0.1 µg/L to 10 µg/L
Second source calibration verification (ICV)	Once after each ICAL.	% recovery $\pm 20\%$	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL	Preferably out-of-house, critiqued standard or else a standard from different lot than calibration standards
Continuing Calibration Verification (CCV)	<u>GC/MS</u> : Daily before sample analysis and every 12 hours of analysis time. <u>GC</u> : Every 10 samples and at end of run	% recovery $\pm 20\%$	Restandardize and rerun all samples from last acceptable QC or check sample	Approximate mid-range standard made from working standards stock
Continuing Calibration Blank (CCB)	Once to check instrument, then after every CCV	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Method Blank (BLK)	Once per run	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank with same reagents as samples
Preparation Blank (BLK)	With each preparation batch	< RL	Correct problem. Reanalyze all samples < 10 times RL value	DI water blank that is treated exactly as a sample including exposure to all glassware, equipment, solvents, and reagents that are used with other sample analysis.
Laboratory Control Sample (LCS) Also known as blank spike (BS)	Once per batch or every 20 samples	Spike recovery within statistical limits from DoD LCS Study	Same as matrix spike	DI water fortified with the same spike as the MS/MSD

QC Check (Symbol)	Run Frequency	Acceptance Criteria	Corrective Action	Comments
Matrix Spike (MS)	Once per batch or every 20 samples	Spike recovery same as LCS	Check for instrument drift, respoke, and retest	Fortified unknown sample
Matrix Spike Duplicate (MSD)	Every MS	Spike recovery same as MS; RPD \leq 30% between MS and MSD	Same as matrix spike	Duplicate MS
Detection Limit Standard (CRL)	Once per run	\pm 50% of expected value	Correct instrument's sensitivity problem or redetermine and raise reporting limits	Low level standard at the RL
Surrogate spike	Every field sample, standard, and QC sample.	% recovery: 60 – 130%	For QC and field samples, correct problem then reprep and reanalyze all failed samples with failed surrogates, if sufficient sample material is available.	If obvious chromatographic interference with surrogate is present, reanalysis is not be necessary.
Standard Reference Material (SRM)	Once per batch (minimum)	N/A		May be used in place of an LCS
Internal standards verification (GC/MS)	Every field sample, standard, and QC sample.	EICP area within \pm 50% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions.	
Tuning (GC/MS)	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify.	

Sample Preparation Summary

Preparation Method: 3520C
Sample Amount: 1000 mL
Final Volume: 1 mL